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MgO micromachining for superconductor focal plane arrays

C. Alain [a], L. Ngo Phong [b], and I. Shih [a]

[a] Electrical Engineering Department, McGill University, Montreal, QC H3A 2A7, Canada
[b] Defence Research Establishment Valcartier, Val-Belair, QC G3J 1X5, Canada

ABSTRACT

The construction of superconductor focal planes for infrared or millimeter wave imaging requires that the substrate of superconductor films be micromachined into thermal isolation structures or horn cavities. Wet etching was used to create cavities in the MgO substrate of high T_c BiPbSrCaCuO films. Processes for lithography of metal patterns on superconductor films were also devised. It was found that cavities with a wall angle of 55 - 60° could be formed in (100) MgO using solutions of HNO_3:CH_3COOH or H_3PO_4. The MgO normal etch rates of these solutions were found to be respectively 117 and 27 μm / hr. Thermal evaporation and magnetron rf sputtering were used to prepare Au and Ag films on BiPbSrCaCuO and MgO; however, only the sputtered films showed adequate film adhesion. Electric contacts and dipoles made of Au or Ag could be created by wet etching in a solution of KI-I without apparent degradation of the superconductivity of BiPbSrCaCuO.

Keywords: Focal planes, micromachining, wet etching, BiPbSrCaCuO, MgO.

1. INTRODUCTION

The application of high T_c superconductors as transition edge bolometers has received much attention in recent years.[1] The multispectral capability of superconductor bolometers, not pertaining to photon detectors, is attractive with regard to extracting and enhancing image contrast. To attain a performance adequate for thermal infrared imaging, it is necessary that the superconductor focal plane be constructed on thermal isolation structures. Such structures can be created in the substrate supporting the superconductor film, provided micromachining of this substrate is achievable.

At millimeter wavelengths, Josephson detectors and mixers made of high T_c superconductors may offer good performance when integrated into a horn antenna structure. High efficiency horn antennas were fabricated for monolithic receivers by micromachining techniques. Pyramidal horn cavities with a wall angle of 35° and gains of ~ 11 to 20 dB were created in Si wafers.[2] The horn focused radiation energy onto a dipole suspended on a micromembrane; the membrane thickness was much smaller than the free space wavelength so that dielectric losses were eliminated. Because the design of horns can be scaled for different wavelengths, imaging arrays may be constructed for use at high frequencies so as to achieve high resolution and small size inexpensively. An analogous approach may be used for the construction of high T_c superconductor focal planes, as shown in Fig. 1. The proposed device is composed of at least two stacked wafers. Horn cavities are created in the MgO front wafer. Reflecting cavities and detection circuits are implemented in the Si back wafer. The use of MgO front wafers enables the direct growth of high T_c superconductor films on the back side of MgO for the fabrication of superconductor planar antennas and sensors.[3] Again, the feasibility of this device relies on the ability to micromachine the substrate supporting the superconductors.

In this work, the micromachining of monocrystalline (100) MgO substrate was investigated for the construction of BiPbSrCaCuO superconductor focal planes. In sections 2 and 3, the use of wet etching is discussed and the effect of several process variables is presented. Because the substrate was subject to significant mechanical stress during the high temperature crystallization of the BiPbSrCaCuO films, it was preferable that the etching of the substrate be carried out after the film was grown on the substrate. As copper oxide supercon-

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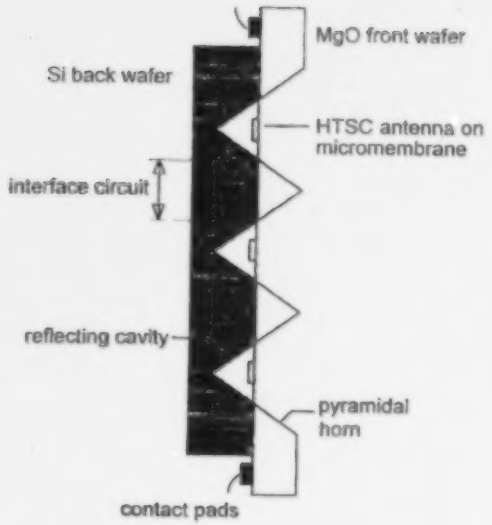

Fig. 1. Cross sectional schematic of a high temperature superconductor (HTS) focal plane.

ductors are erodible in most etching solutions for MgO, the superconductors need to be protected during the etching of the substrate. Lithography processes were devised to create and remove metal patterns on BiPbSrCaCuO films without degrading the film superconductivity. The results obtained for these processes are presented and discussed in section 4.

2. EXPERIMENTAL APPROACH

In view of constructing superconductor focal planes, approaches to micromachining of the supporting substrate of superconductor film were examined. Wet and dry etching methods have been developed for use in micromechanical structures. The wet etching method is inexpensive and provides selectivity, uniformity, and immunity to high energy and high temperature effects. However, wet etchants often dissolve the photoresist. Thus, masking films of a more resistant material may be necessary. The dry etching methods include plasma etching, reactive ion etching, and magnetron ion etching. These methods enable photoresist masking and are better suited for streamlined anisotropic etching, but at the cost of expensive facilities. In addition, because it is more difficult to achieve selectivity and uniformity by means of dry etching, a great care is required to control all the process variables. Other etching methods such as combustion flame and laser ablation may be used. However, the former method restricts the choices of masking materials and the latter asks for costly equipment. Within the scope of this study we consider only the application of wet etching and its effects on the normal and lateral dissolution of (100) oriented MgO or $LaAlO_3$ substrates.

The preferred etching solutions for water insoluble ionic crystals, such as MgO, are acids and alkalis. As shown in Table I, the etch rate and dissolution form depend on many internal and external factors. When the substrate is wet etched, the etchant breaks the bonds between atoms that compose its surface. The etch rate follows the density of kinks in the crystal. This density of kinks changes with the crystal planes, resulting in an anisotropic dissolution. At the interface between the crystal and the solvent, a double layer[4] is created because of the difference in chemical potential of the ions in the crystal and in the solution. When the solution is stagnant, the thickness of the interfacial layer will be the same for all the surfaces in contact with the solvent. This produces an artificially high and uniform density of kinks, resulting in a more isotropic dissolution. Hence, it is important that the etching solution be stirred to achieve anisotropic etching. Agitation can enhance the preferential adsorption of solvent on some planes of the substrate, leading to a different etch rate for each crystal plane.

Internal factors (relative to the crystal)	External factors (relative to the solvent)
- chemical nature	- chemical nature
- type of bonding	- composition
- presence of impurities	- temperature
- crystallographic orientation	- agitation
- lattice imperfection	- impurities
- cracks	- etching time

Table I. Variables affecting the dissolution of a single crystal.

Because of the dislocations and other defects normally present in the crystalline substrate, it is relatively difficult to evenly etch these substrates in wet solutions. Etch pits or small pyramids tend to form as the area surrounding a dislocation is etched away more rapidly. There appears to be no effective treatment for the problem of crystal imperfection. A pre-etch heat treatment of the MgO substrate may reduce the defect density but promote the formation of large surface steps.[5] Therefore, the use of high quality substrates is important in order to achieve a more uniform dissolution.

H_3PO_4, HCl, and HF have been used as etching solutions for monocrystalline MgO. To construct pyroelectric infrared sensors, Kotani *et al.* used 10 % vol. H_3PO_4 pre-heated to 80 °C to form lozenge micro cavities in MgO.[6] The MgO etch rates in 0.74 M H_3PO_4 at 65 °C were reported to be 55 μm / hr in the (100) plane and 37.5 μm / hr in the (110) and (111) planes.[4] The cavity walls formed an angle of 34° with the normal of (100) plane. To polish the MgO surface, Hadara suggested stirring of the MgO sample in boiling 85 % vol. H_3PO_4 for several seconds.[7] Heimann *et al.* proposed the dissolution of (100) MgO using 4.5 N HCl at 100 °C.[8] Sugiura *et al.* reported a normal etch rate of 96 nm / hr of MgO in 36 wt % HCl at 20 °C.[9] The lateral dimension of the etch step was in the range of 4 to 5 μm regardless of the duration of the dissolution; hence, the achieved angle of the wall was a function of the etch depth. These authors suggested that HCl is a better solution than H_3PO_4 with regard to the surface polishing of MgO. The temperature dependence of the lateral etch rate in HCl was specifically investigated by Sangwal.[4] It was shown that the lateral etch rate of MgO in 2 N HCl increased from 22 to 400 μm / hr when the temperature of HCl was increased from 29 to 80 °C. Finally, the use of HF etchant for MgO was investigated by Eidelloth *et al.*.[10] These authors reported an etch rate in the range of 4.8 - 9.6 μm / hr for 7 % HF at 23 °C.

Little information has been reported on the wet etching of monocrystalline $LaAlO_3$ in literature. In a preliminary experiment, we have investigated the effect of H_3PO_4 and HF etchants on (100) $LaAlO_3$ substrates. The measured etch rates for these etchants were small, suggesting that the wet etching method may be unsuited for use on $LaAlO_3$. As a result, the effect of etching solutions was investigated for MgO substrates only.

3. MICROMACHINING OF CAVITIES IN MgO SUBSTRATE

A systematic study of the effect of etching solutions on the MgO substrate was carried out with the idea of micromachining the horn cavities in this substrate. The etchant to be selected is subject to specific criteria. It is imperative that the etched walls be even and form an angle of 30° to 60° with respect to the normal of the (100) MgO plane. Furthermore, the etchant must produce a MgO etch rate high enough to be of practical use. The etchants examined in this study included H_3PO_4, HCl, H_2SO_4, HNO_3, HF, buffered HF, acetic acid, and acetyl acetone. The concentration and temperature of the etchant were used as the process variables. We also observed a dependence of the etch rate on the concentration of water in the etching solutions and on the application of etchant agitation. The experimental details and results of this study are reported as follows.

3.1 Experimental

Monocrystalline (100) MgO substrates were cleaned in acetone, rinsed with 2-propanol, and spin dried. The area of the substrate was typically 5 x 5 mm^2. An Au film with a thickness of ~ 1 μm was deposited on top of the substrate, serving as a mask. The deposition of the Au film was performed by magnetron rf sputtering in 1 mTorr of Ar at a rate of 1 μm / hr. Under these conditions, the deposited film was seen to adhere strongly to the substrate. Standard photolithography was used to open square windows in the Au mask; in this process a KI-I solution was used to remove the unwanted area of the Au film. After the remaining photoresist on the film was removed, the sample was immersed in the etching solution. Temperature control of the solution was achieved by means of a thermometer, temperature controller, and a heater in a feedback loop.

The choice of Au over the photoresist for the substrate masking was primarily due to the rapid dissolution of photoresist in the studied etchants. Au was found to be resistant to these etchants in the range of etchant concentrations used in the experiment. Furthermore, as discussed in section 4, a lithography process was established to create Au patterns on superconductor BiPbSrCaCuO without degrading its superconductivity.

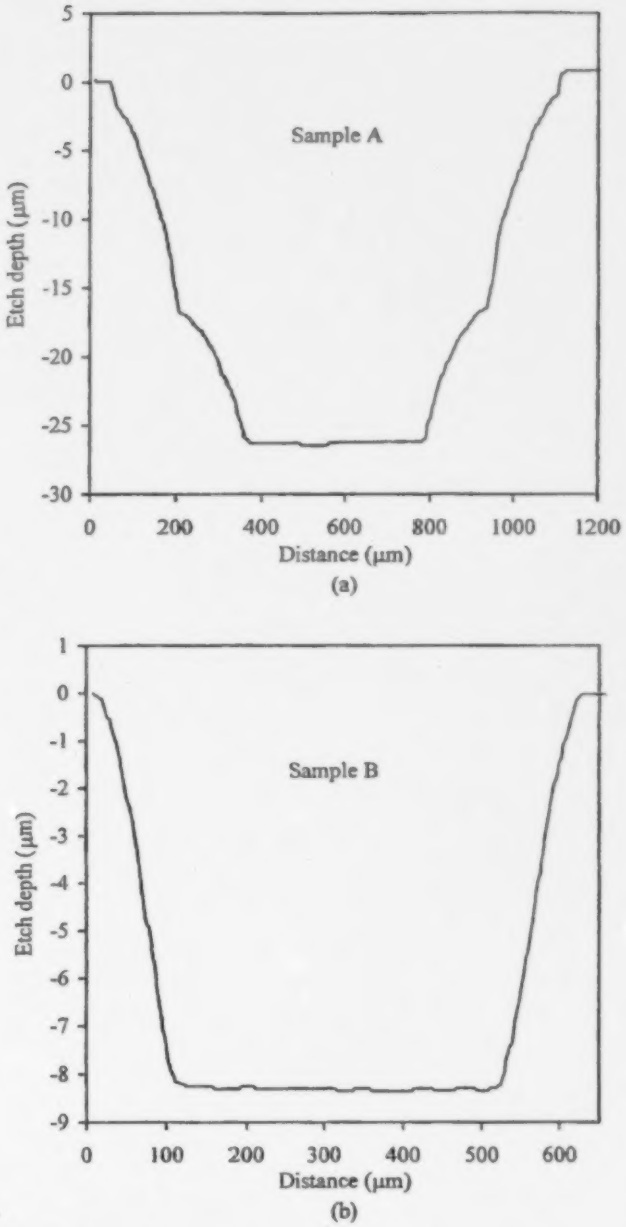

Fig. 2. Typical surface profile of MgO cavities resulted from etching in: (a) a water based solution; (b) a methanol based solution.

Therefore, the Au film can further be used as a protective coating for the superconducting film during the wet etching of the MgO substrate.

3.2 Effect of water based etching solutions

Because the MgO substrate is hygroscopic, the presence of water in an etching solution may affect the etch rate of the solution and the geometry of the resulting cavity. To investigate the effect of water, two different etching solutions were prepared. The first solution consisted of 10 % vol. H_3PO_4 in de-ionized water. The second solution consisted of 10 % vol. H_3PO_4 in methanol. The temperature of both solutions was maintained at 60 °C during the experiment. Figure 2 shows the surface profiles of two MgO samples, sample A being etched in the first solution and sample B being etched in the second solution. Both samples were etched for two periods of 5 min, separated by a short period in which sample A was rinsed with water and sample B with methanol. After this, the samples were spin dried. It can be seen in Fig. 2 that the MgO etch rate in the water based solution is about three times higher than that in the methanol based solution. This result is in agreement with the theoretical prediction[4] that the etch rate is 1.2 to 6 times lower when MgO is unhydrated. The water molecules adsorbed on the substrate appeared to ease the dissolution, but also to create irregular geometry. The surface profile of the cavity etched in the water based solution shows varying etch rates and a visible transition between the etching periods. In contrast, the surface profile associated with the methanol based etchant reveals uniform etch rates and a transparent transition between etching periods. In the absence of water in the etching solution, the resulting walls of the cavity were smooth. Both solutions resulted in a wall angle of ~ 85° with respect to the normal of the (100) plane of MgO.

3.3 Effect of etchant agitation

To confirm the effect of etchant agitation, we have measured the surface profiles of the cavities etched in both stagnant and stirred solutions. Figure 3 shows the profile of a cavity etched in a stagnant solution of H_3PO_4 and 2-propanol. It is believed that the rounded bottom surface of this cavity was due to the higher concentra-

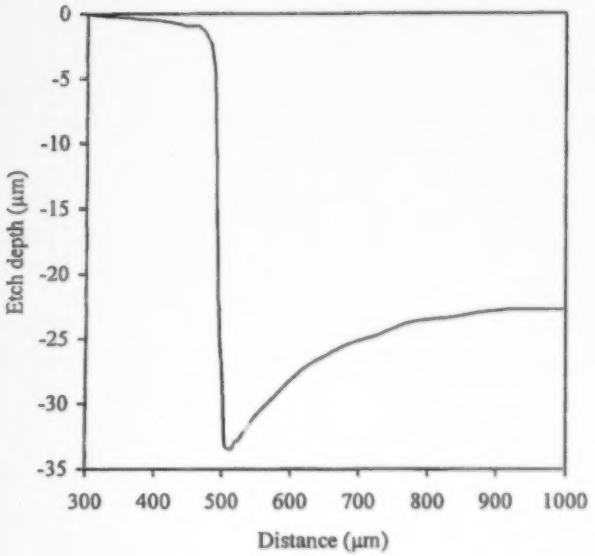

Fig. 3. Surface profile of a MgO cavity resulted from etching in a stagnant solution of H_3PO_4.

tion of etchant near the edges of the cavity. This fact may arise from the smaller density of dissolved molecules near the edges in a stagnant etchant. In contrast, the profile of the cavity etched in a stirred solution showed an even bottom surface (see, for example, Fig. 2*b*). The observed effect of etchant agitation on anisotropic etching was consistent with the discussion outlined in section 2.

3.4 Effect of concentration and temperature

To determine the etching solutions and conditions suited for the creation of horn cavities, the dissolution of MgO substrate was examined for different concentrations and temperatures of the etchants. All etching solutions were prepared in 2-propanol solvent and stirred during the experiment. The principal results are summarized in Table II. It follows from these results that both the HNO_3:CH_3COOH and 25 % vol. H_3PO_4 solutions may be adequate for the etching of MgO. The application of the former solution resulted in horn cavities with a constant wall angle of 60° and smooth wall surfaces. When the temperature of this solution was increased from 50° to 70°, the normal etch rate increased from 26 to 117 μm / hr. The dissolution of MgO in low concentration solutions of H_3PO_4 resulted in uneven wall surfaces. However, when the acid concentration was increased to 25 %, a polished wall surface was obtained. In addition, the normal etch rate of MgO in 25 % vol. H_3PO_4 (~ 27 μm / h at 70 °C) was sufficiently high for the practical use of this solution. Sangwal[4] attributed the surface polishing action of H_3PO_4 at high concentrations and elevated temperatures to a strong adsorption of acid molecules and reaction products on the etched surfaces.

The main difficulties encountered when applying the other etchants were the resulting rough surfaces, irregular geometries, and low etch rates. The dissolution of MgO in dilute or pure HCl tended to produce, respectively, irregular steps in the etched wall or circular cavities. The surfaces etched in H_2SO_4 were uneven, as possibly due to the insoluble nature of phosphates of magnesium.[4] Finally, it was found that little practical use could be made of HF, buffered HF, and acetyl acetone because of the extremely low MgO etch rates in these solutions.

4. LITHOGRAPHY OF METALS ON SUPERCONDUCTOR FILMS

Copper oxide superconductors are erodible in acid solutions. The use of wet etching in the fabrication of superconductor focal planes makes it imperative that the superconductor sensor be shielded during the etching of the substrate. Noble metals, such as Au, are known for their resistance to acid solutions and may be candidates for the shielding of superconductor. In this work, processes to deposit and remove Au patterns on superconductor BiPbSrCaCuO were investigated. Once determined, these processes may also be used in the metallization and fabrication of metal probe dipoles and contacts on the superconductor focal plane. Since it is preferable that the superconductivity of the sensor remain unaffected by these processes, the superconducting properties of BiPbSrCaCuO before and after the lithography of Au were studied and compared.

Etching solution	*Temperature* (°C)	*Etch rate* (μm / hr)	*Characteristics*
5 % vol. H_3PO_4	20	0.38	etched surface was rough for low concentrations or temperatures; pyramidal cavities are with angles of ~ 55°
	25	0.8	
	30	0.87	
	50	0.58	
	70	0.17	
10 % vol. H_3PO_4	25	0.76	
20 % vol. H_3PO_4	25	1.07	
	50	32	
	70	35	
25 % vol. H_3PO_4	20	2.0	
	30	2.1	
	50	23	
	70	27	
30 % vol. H_3PO_4	25	3.1	
40 % vol. H_3PO_4	25	3.9	
20 % vol. H_2SO_4	50	0.28	bumps of ~ 6 μm on the (100) plane
	70	0.77	
1:1 vol HNO_3 : CH_3COOH	50	26	smooth wall surfaces with a constant angle of 60°
	70	117	
20 % vol. HCl	30	0.6	irregular steps
	50	3.1	
	70	23	
100 % vol. HCl	17	0.4	circular cavities with smooth surfaces
	50	9.4	
	70	32	
20 % vol. HF	17	0.06	etched surfaces of dry skin type
Buffered HF (with water)	17	0.04	low etch rate
40 % vol. acetyl acetone	70	0.02	

Table II. MgO normal etch rates for various concentrations and temperatures of etching solutions. Unless specified otherwise, the etching solutions were prepared in 2-propanol solvent.

4.1 Deposition of Au on superconductor film

The BiPbSrCaCuO superconductor films used in this experiment were prepared on (100) MgO substrates by magnetron rf sputtering. No intentional heating was applied to the substrate during the deposition. The film thicknesses were between 20 and 300 nm. After a short heat treatment, first in oxygen and then in air, nearly single crystal superconducting films with T_c's above 100 K resulted. Further details on the preparation and properties of these films were reported elsewhere.[3]

In the first experiment, thermal evaporation of high purity Au pellets was carried out at a pressure of 10^{-6} Torr to prepare Au films on BiPbSrCaCuO. Approximately 400 mg of Au was necessary to achieve films with a thickness of ~ 800 nm. Under these conditions, it was found that the deposited film adheres weakly to the superconductor film. In effect, Au films tended to peel off when submitted to the mechanical stress induced either by thermosonic wire bonding or reaction in acid solutions. Similar results were noted when the Au film was deposited on the (100) MgO substrate under the same conditions. To verify whether the poor adhesion of Au films resulted from contamination or imperfection of the substrate surface, the substrate was thoroughly cleaned in hot H_3PO_4 and heat treated at 500 °C for 10 min prior to the deposition. Despite this additional preparation, the adhesion of Au film on MgO did not improve.

In the second experiment, magnetron rf sputtering was used to deposit Au films on both BiPbSrCaCuO and (100) MgO. The deposition was performed in 1 mTorr of Ar at a rate of ~ 1 μm / hr. The Au films were prepared with a thickness in the range of 0.2 to 1 μm. Unlike the evaporated film, the sputtered film showed a

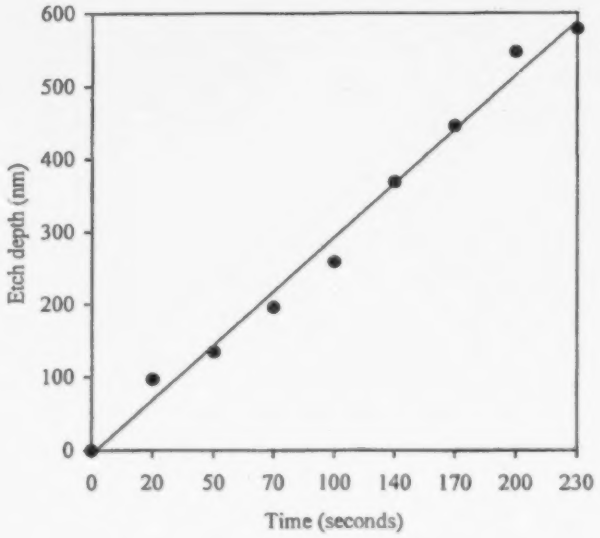

Fig. 4. Etch depth of Au film as a function of time in a KI-I solution; the solid line depicts a slope of 9 μm / hr.

better adhesion to both BiPbSrCaCuO and MgO substrates. After the deposition, the film underwent successfully the wire bonding and chemical soaking tests. The better adhesion of sputtered Au films, as compared with evaporated Au films, might be due to the higher energy of the sputtered Au atoms incident onto the substrate. Another possibility is that the substrate bombardment by secondary target electrons during the sputter deposition may have promoted interfacial reaction and interdiffusion in the substrate.

4.2 Wet etching of Au films

The inadequacy of the thermal evaporation of Au prevents the use of lift off technique to create metal patterns on the superconductor film. An alternative, devised by Eidelloth and Sandstrom[11], enabled wet etching of dc sputtered Au films on superconductor YBaCuO. These authors reported an Au etch rate of 1.54 μm / hr in a solution consisting of 4 g KI, 1 g I and 150 ml de-ionized water. In addition, the superconductivity of YBaCuO films was unchanged after the patterning. In this work, the applicability of the KI-I solution as an etchant for the Au protective film for the BiPbSrCaCuO superconductor was investigated. In view of obtaining a higher Au etch rate, we examined the use of KI-I solutions with concentrations higher than that employed for YBaCuO.

In the first experiment, the Au etch rate was measured for various concentrations of the KI-I solution. The Au film sample, having a thickness of 800 nm, was sputter deposited on the MgO substrate. After the deposition, the Au film was masked with positive photoresist and a window was opened in the photoresist. Once the sample was prepared, it was immersed in the KI-I solution, rinsed with de-ionized water, and spin dried. Figure 4 shows the time variation of the etch depth in a stirred solution of 4 g KI, 2 g I and 80 ml de-ionized water. The temperature of the etching solution was maintained at 20 °C. It is seen from the slope of the curve in Fig. 4 that the average Au etch rate was 9 μm / hr, which is 6 times the etch rate reported by Eidelloth and Sandstrom.[11] The etch rate decreased as the solution aged and became saturated with the etched particles. It was also noted that the lateral etch rate was almost similar to the normal etch rate. It follows that the KI-I solution may be unsuited for use in the precision patterning of thick Au films.

The second experiment was carried out to determine the effect of the employed KI-I solution on the superconductivity of the BiPbSrCaCuO film. In this experiment, the resistance-temperature (R-T) characteristic of several BiPbSrCaCuO films grown on MgO substrates and patterned into four point probes was first measured. Next, Au was sputter deposited on top of the superconducting films. The Au shielded films were etched in the KI-I solution until the films were exposed, etched further for a period of 5 min, rinsed with de-ionized water, and spin dried. Figure 5 shows the R-T characteristic of an etched film, which is seen to be identical to the characteristic measured prior to deposition and etching of Au. In addition, optical microscopy examination of the etched films did not reveal any apparent defects on these films. In light of these results, it appears that the superconductivity of BiPbSrCaCuO was unaffected by the proposed etching solution for the Au films.

4.3 Lithography of other metals

Once the process for the lithography of Au was established, we further examined the applicability of this

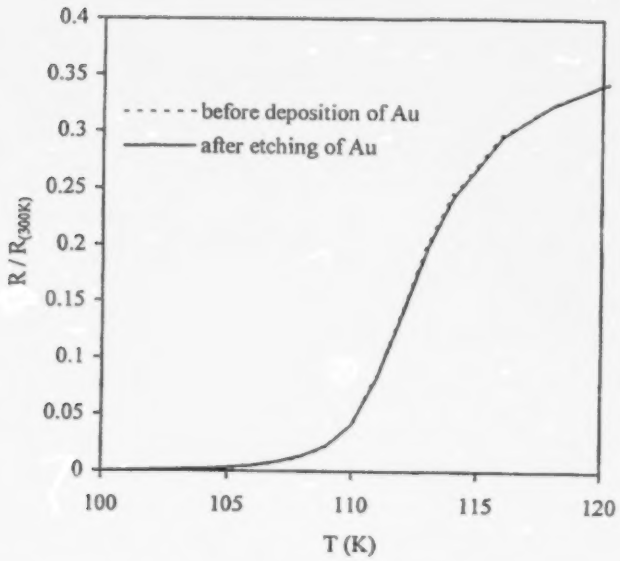

Fig. 5. *R-T* characteristics of BiPbSrCaCuO films before the deposition and after the etching of Au films.

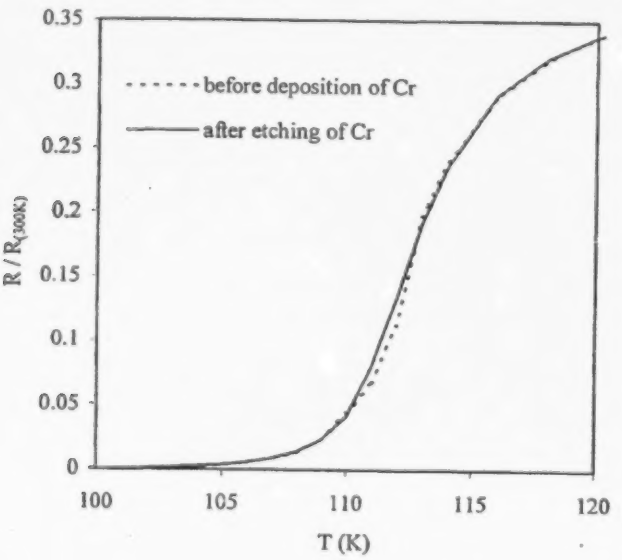

Fig. 6. *R-T* characteristics of BiPbSrCaCuO films before the deposition and after the etching of Cr films.

process to other metals, such as Ag and Cr. It was found that the process devised for Au produced similar results when applied to the lithography of Ag films. Unlike Ag, the electrical conductivity of Cr is too low for the use of Cr as electrical contacts or dipoles on BiPbSrCaCuO films. However, Cr is inexpensive and may be a candidate for the masking of the MgO substrate during the fabrication of cavities in the substrate. Furthermore, evaporated Cr films appeared to adhere strongly to the superconductor film. In this work, thermal evaporation of a Cr plated tungsten rod was performed to deposit 200 nm of Cr on BiPbSrCaCuO film. The wet etching of Cr was carried out in a solution composed of 4 g of $KMnO_4$, 50 ml of 0.1 N Na H, and 50 ml of de-ionized water, using the same techniques as described previously. The Cr etch rate in this solution was ~ 18 μm / hr. In Fig. 6, the *R-T* characteristic of the etched film is seen to differ only slightly from the characteristic recorded before the etching. This result indicates that the effect of the etching solution for Cr on BiPbSrCaCuO superconductor may be negligible. The dissolution of Cr using the proposed etching solution has one disadvantage. Due to the presence of NaOH in the solution, the masking photoresist reacts with the solution and is dissolved, leaving residues on the Cr film to be etched.

5. CONCLUSIONS

The construction of superconductor focal planes for infrared or millimeter wave imaging requires that the substrate of superconductor films be micromachined into thermal isolation structures or horn cavities. In this work, the use of wet etching has been investigated in order to create cavities in the MgO substrate of high T_c BiPbSrCaCuO film. The dependence of the MgO etch rate on the concentration of water in etching solutions and on the etchant agitation was first examined. After this, it was found that cavities with a wall angle of 55-60^0 could be formed in (100) MgO using solutions of HNO_3:CH_3COOH or H_3PO_4. The MgO normal etch rates of these solutions were measured and found to be respectively 117 and 27 μm / hr. Because BiPbSrCaCuO superconductor is erodible in these solutions, processes for the lithography of metal patterns on superconductor film were also devised. Thermal evaporation and magnetron rf sputtering were used to pre-

pare Au and Ag films on BiPbSrCaCuO; however, only the sputtered films showed adequate film adhesion. The wet etching of metals in a solution of KI-I that followed showed that Au or Ag masks, contacts, dipoles, and other patterns could be formed without apparent degradation of superconductivity of BiPbSrCaCuO.

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PERSONAL AUTHOR: Alain, C.; Ngo Phong,L.; Shih, I.

CORPORATE AUTHOR: Defence Research Establishment Valcartier, Valcartier QUE (CAN)

ABSTRACT: (U) The construction of superconductor focal planes for infrared or millimeter wave imaging requires that the substrate of superconductor films be micromachined into thermal isolation structures or horn cavities. Wet etching was used to create cavities in the MgO substrate of high Te BiPbSrCaCuO films. Processes for lithography of metal patterns on superconductor films were also devised. It was found that cavities with a wall angle of 55 - 60 degrees could be formed in (100) MgO using solutions of HN03:CH3C00H or H3PO4. The MgO normal etch rates of these solutions were found to be respectively 117 and 27 mu m/hr. Thermal evaporation and magnetron rf sputtering were used to prepare Au and Ag films on BiPbSrCaCuO and MgO; however, only the sputtered films showed adequate film adhesion. Electric contacts and dipoles made of Au or Ag could be created by wet etching in a solution of KI-I without apparent degradation of the superconductivity of BiPbSrCaCuO.

DESCRIPTORS: (U) Focal plane arrays;Infrared equipment;Microwave equipment;Millimeter waves;Superconductors;Infrared detectors;Electric fields;Etching;Sputtering;Magnetic storage;Thin films;Substrates;Superconductivity

IDENTIFIERS: (U) Micromachining;Josephson detectors

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